



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/US92/07456 <b>(22) International Filing Date:</b> 1 September 1992 (01.09.92)  <b>(30) Priority data:</b> 756,610                      9 September 1991 (09.09.91)    US 911,164                      9 July 1992 (09.07.92)            US  <b>(71) Applicant:</b> AMERICAN TECHNOLOGY, INC. [US/ US]; 1402 Schillington Drive, Katy, TX 77450 (US).  <b>(72) Inventors:</b> KOCH, William, J. ; 616 Ridgeway Drive, Lib- erty, MO 64068 (US). CANNON, Collins, P. ; 16323 Plattsburg, Kearney, MO 64060 (US).  <b>(74) Agent:</b> MORGAN, Terry, D.; P.O. Box 4433, Houston, TX 77210 (US).		<b>(81) Designated States:</b> AT, AU, BB, BG, BR, CA, CH, CS, DE, DK, ES, FI, GB, HU, JP, KP, KR, LK, LU, MG, MN, MW, NL, NO, PL, RO, RU, SD, SE, European pa- tent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> SPINEL INSULATED ELECTRICAL COMPONENTS AND METHOD FOR MAKING SAME		
<div style="text-align: center;"> </div>		
<b>(57) Abstract</b> <p>A mineral insulated electrical component, such as a cable (18) or sensor, has a center conductor (12), a metallic sheath (16), and ceramic insulation formed from spinel (<math>\text{MgAl}_2\text{O}_4</math>) disposed therebetween. The spinel insulated component possesses high resistance to environmental stresses, such as high temperature, pressure, long term hard neutron radiation, and corrosive chemical environments.</p>		

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SPINEL INSULATED ELECTRICAL COMPONENTS  
AND METHOD FOR MAKING SAME

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This invention is generally directed to mineral insulated electrical components, such as cables and sensors, and, more particularly, to spinel insulation used in such cables and sensors, methods for producing the spinel insulation, a spinel preform, and a spinel insulated cable.

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Mineral insulation is often used in the fabrication of electrical components, such as sensors and cables. Mineral insulation is known to provide good resistance to environmental stresses, such as thermal and nuclear radiation. Heretofore, significant shortcomings have existed in the use of conventional mineral insulation. These shortcomings are evident in the problems associated with mineral insulated cables, but exist in many other types of electrical components, such as sensors. Accordingly, while the problems described below are illustrated in conjunction with mineral insulated cables, they are common to most other applications of mineral insulation, including sensors.

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Typically, a hard cable includes an inner conductive wire surrounded by a mineral insulator, such as a ceramic, and placed within a metallic jacket. The mineral insulator resists the effects of environmental

invasive stress, such as radiation, owing to its crystal structure.

Mineral insulation for cables has typically been  
5 formed from ceramics, such as magnesium oxide (MgO) or  
aluminum oxide (Al<sub>2</sub>O<sub>3</sub>). Both of these ceramics, and  
especially magnesium oxide, have very good insulation  
resistance (if pure) at temperatures to 800°C. Above this  
10 temperature, magnesium oxide tends to react with the  
jacket and/or the internal wire and will form white smoke  
and dissociate in a reducing atmosphere. Aluminum oxide  
does not typically have resistivity values as high as  
those of magnesium oxide at elevated temperatures.  
Further, neither of these materials are effective  
15 insulators over time in a fast neutron radiation  
environment, due to a complete break-down or collapse of  
their crystal structure.

Reaction of the magnesium oxide with the jacket and  
20 internal wire is a clear cause of reduced lifespan of  
magnesium oxide insulated cables. Ordinarily, as an  
oxide, magnesium oxide has a tendency to react with the  
jacket and internal wire; however, when heated, this  
problem is exacerbated, as the heat partially reduces the  
25 magnesium oxide, and the resulting magnesia is highly  
reactive with the internal wire and jacket. Some relief  
has been obtained by venting the jacket of the cable to  
expose the magnesium oxide to the external atmosphere.  
This venting has a tendency to keep the magnesium oxide  
30 in its oxide form, and thereby reduce reaction with the  
jacket and internal wire.

Unfortunately, magnesium oxide has a strong tendency  
to hydrate or absorb water, which necessarily results in  
35 corrosion of the jacket and internal wire. Thus, while  
venting the jacket tends to limit reduction of the

magnesium oxide, and reduce reaction between the magnesium oxide, jacket, and internal wire, it also induces a different type of reactive influence, corrosion. Moreover, hydration reduces the effectiveness of the magnesium oxide as an electrical insulator, and may lead to voltage breakdown between the jacket and wire. These areas of voltage breakdown cause failure of the cable.

10           The present invention is directed to overcoming or minimizing one or more of the problems discussed above.

          In one aspect of the present invention, ceramic insulation in a mineral insulated electrical component is provided. The insulation is comprised of spinel.

          In another aspect of the present invention, a paste suitable for firing into a ceramic for use as insulation in a mineral insulated electrical component is provided. The paste is formed from spinel powder and a binder solution.

          In still another aspect of the present invention, a method for forming a ceramic to be used as insulation in a mineral insulated electrical component is provided. The method includes the steps of: preparing a binder solution; preparing a spinel powder; combining the spinel powder and binder solution into a paste; and firing the paste.

          Other objects and advantages of the invention will become apparent upon reading the following detailed description and upon reference to the drawing in which:

35           Fig. 1 illustrates a perspective cut-away view of a coaxial mineral insulated cable;

Fig. 2 illustrates a perspective cut-away view of a triaxial mineral insulated cable; and

5 Fig. 3 illustrates a cross-sectional view of a coaxial mineral insulated thermocouple.

While the invention is susceptible to various modifications and alternative forms, specific embodiments thereof have been shown by way of example in the drawings  
10 and will herein be described in detail. It should be understood, however, that this specification is not intended to limit the invention to the particular forms disclosed herein, but on the contrary, the intention is to cover all modification, equivalents, and alternatives  
15 falling within the spirit and scope of the invention, as defined by the appended claims.

Referring now to the drawings, and, in particular, to Fig. 1, there is shown an exploded perspective view of  
20 a ceramic insulation 10, a center conductor electrical wire 12, and a metallic sheath 16, which, when assembled, form a mineral insulated or hard cable 18. The metallic sheath 16 is preferably formed from a nickel iron chrome alloy, or a more refractory metal, if required. The  
25 electrical wire 12, is formed from any suitable conductive material, however, copper is preferred. The ceramic preform 10 is preferably formed from a ceramic mixture having a ratio of 28.2% magnesium oxide to 71.8% aluminum oxide by weight, and being fired into new  
30 compound, spinel ( $MgAl_2O_4$ ).

The hard cable 18 represents a first embodiment of the instant invention, with other embodiments, such as a sensor, illustrated and discussed subsequently herein.

The ceramic insulation 10 can be deposited in the metallic sheath 16 using a variety of processes without departing from the spirit and scope of the instant invention. Two preferred processes are described subsequently herein. The first process involves firing the spinel into ceramic preforms and threading the preforms onto the wire 12. Alternatively, the insulation 10 can also be placed in the sheath 16 in powdered or granulated form, using conventional loading, vibrating, and ramming techniques to ensure that voids are not created.

The specific compound and method used in preparing the insulation 10 and depositing it within the sheath 16 are discussed in greater detail below. However, for a proper understanding of the instant invention, it is useful to first appreciate the construction and assembly processes of a hard cable.

In a first method of constructing the hard cable 18, the wire 12 is manually threaded through a series of relatively short preforms 10 (i.e., typically two to four inches each) until a desired length is reached (i.e., typically forty feet). Thereafter, the wire 12 and preforms 10 are inserted into the sheath 16 to form the hard cable 18.

At this point, the assembled hard cable 18 is passed through conventional metallic drawing dies. The drawing dies forcibly reduce the diameter of the sheath 16, and thereby lengthen the cable by a small amount. Thus, by passing the cable 18 through a series of progressively smaller drawing dies, the cable 18 is gradually reduced in diameter and increased in length until a desired diameter and/or length is reached. It is preferable to

heat treat (anneal) the cable between successive draws to remove work hardening from the metal sheath 16.

5 It should be appreciated that the ceramic preforms 10 positioned within the cable 18 are relatively hard, but are crushed during the drawing or swaging operation. This crushing action, however, advantageously allows the ceramic to fill any voids within the cable 18 and thereby improve the cable's electrical properties.

10 Thermocouples and small diameter mineral insulated electric cables are commonly constructed using the above-described ceramic preform technique. Mineral insulated heaters, thermocouples, and many types of electrical  
15 cables are, however, commonly constructed using a powder fill method, as described below.

In the powder fill method, the spinel insulation 10 is reduced to or obtained in a powdered or granulated  
20 form and then loaded into the sheath 16 using conventional techniques. These conventional techniques are in common usage to produce MgO type insulated cables, and, therefore, need not be discussed in detail herein. For purposes of understanding this invention, it is  
25 sufficient to recognize that the powdered spinel is typically placed in the sheath 16 about the wire 12 by a mechanism such as a funnel loading or screw type auger as the sheath 16 is vibrated. Vibration aids in compacting the powder and prevents formation of voids in the  
30 insulation 10. Ordinarily, the powder is also tamped into the sheath during this process. In most cases it is desirable to swage or draw down the cable 18 to further compact the spinel to enhance the electrical characteristics of the cable.



The instant invention also finds beneficial application in multi-conductor cables, i.e. cables where the number of electrical conductor wires present within the interior of the ceramic insulation varies from two to upwards of 200. In cable geometries of this type, it is generally preferable to position the wires so that insulation thickness between adjacent wires and between wires and the sheath are equal.

The instant invention also finds beneficial application in triaxial-type hard cables. Triaxial-type hard cables are substantially similar to that shown in Fig. 1, but include an additional metallic sheath 20 positioned between the wire 12, and the sheath 16 (see Fig. 2). Insulation 22, 24 is provided between the sheaths 16, 20 and also between the additional sheath 20 and the wire 12. In such a triaxial-type hard cable, the insulation 24 placed between the wire 12 and additional sheath 20 is preferably spinel, whereas the insulation 22 placed between the sheaths can take any of a variety of forms, including, but not limited to, mineral insulators, such as spinel.

Fig. 3 illustrates a slightly different use of the spinel insulator 10 from the above-described use in cables. That is, the insulator 10 is useful in not only cables, but also in electrical sensors, which may be attached to the cable 18. Hardened sensors are useful for the same reasons that hardened cables are useful in environmentally stressful conditions. In fact, attaching a non-hardened sensor to a hardened cable may not extend the useful life of the system, since the sensor remains susceptible to early failure.

While many types of sensors are envisioned as being benefited by the use of spinel insulation, its

application to thermocouples is illustrative of the application to sensors in general. Fig. 3 illustrates a cross sectional view of a thermocouple 50 formed from an outer sheath 52, a pair of internal dissimilar wires 54, 56, and a mineral insulator 58.

The methods useable for constructing the thermocouple 50 are substantially identical to those discussed above in conjunction with the cable 18. Further, the methods described below for constructing the spinel insulation are equally useful in constructing the mineral insulator 58.

It is also envisioned that some applications of mineral insulation to sensors, and even cables in some instances, do not require crushed or powdered insulation. Rather, some electrical components can make use of the ceramic preform 10 in its hard body configuration.

Spinel ( $\text{MgAl}_2\text{O}_4$ ) formed from a one to one molar ratio of magnesium oxide ( $\text{MgO}$ ) and aluminum oxide ( $\text{Al}_2\text{O}_3$ ) exhibits superior resistance to the effects of fast neutron radiation. It also exhibits superior stability when subjected to temperature extremes, which are a common projection for many accident scenarios, such as might occur in a nuclear reactor.  $\text{MgAl}_2\text{O}_4$  overcomes the limitations inherent when  $\text{MgO}$  and  $\text{Al}_2\text{O}_3$  are used alone as the insulation in a hard cable.

Spinel's resistance to radiation and temperature extremes is due to its unique crystal structure. Spinel has a basic cubic structure. This cubic structure is so fundamentally stable that if long term hard radiation knocks out one or two of the magnesium or aluminum cations, the structure does not collapse. X-ray analyses of this structure does not reveal any differentiation of

the oxygen atom arrangement in the lattice, nor any special coordination of the magnesium to a particular pair of oxygen atoms, or of aluminum to an arrangement of oxygen atoms. All oxygen atoms (ions) in the lattice are  
5 equivalent, forming a close-packed cubic structure.

The elementary cell of spinel contains eight "molecules" of  $\text{MgAl}_2\text{O}_4$ , or eight magnesium ions, sixteen aluminum ions, and thirty-two oxygen ions, for a total of  
10 fifty-six ions. Since the structure of  $\text{MgAl}_2\text{O}_4$  is determined by the space configuration of comparatively large oxygen ions, between which small tri and bivalent cations are inserted, a material exchange and even a certain disorder of the cations is possible due to  
15 neutron radiation, without causing a basic change in the lattice.

$\text{MgAl}_2\text{O}_4$ , therefore, because of its stoichiometry and extremely stable crystal structure, provides superior  
20 resistance to degradation of its dielectric and physical properties under the separate or combined environmental stresses of elevated temperature and nuclear radiation.  $\text{MgAl}_2\text{O}_4$  does not, therefore, react with metals, as do  $\text{MgO}$  and  $\text{Al}_2\text{O}_3$ .

25

An obstacle to fabrication of a metal sheathed electrical cable using  $\text{MgAl}_2\text{O}_4$  as the insulation material has been the unavailability of a spinel crushable preform. Crushable spinel preforms have not been  
30 available, commercially or otherwise, for fabrication of a metal sheathed,  $\text{MgAl}_2\text{O}_4$  insulated cable.

The method for fabricating the spinel preform begins with obtaining starting materials that are in a usable  
35 condition. The need to place the starting material used to fabricate the preform in a "desired" stoichiometric

state is of primary concern. To accomplish this, it is preferable that an X-ray diffraction pattern of the "as-received" materials is obtained. A subsequent analysis of the X-ray diffraction pattern is then performed to  
5 establish the "as-received" stoichiometric state and to assure that complete reaction of all alumina or magnesia grains has been achieved to form the approximately 1:1 molar  $\text{MgO-Al}_2\text{O}_3$  compound. If any portions of the alumina or magnesia grains remain unreacted, or impurities are  
10 present, then the required stoichiometry is not present, and a "firing schedule" should be established to place the spinel material into a desired stoichiometric state. It is the presence of the desired stoichiometric state that gives the final spinel insulated cable its  
15 extraordinary stability under environmental stresses such as radiation, temperature, pressure, etc.

Another obstacle to the formation of a satisfactory preform is the control of trace contaminants.  
20 Contaminants in the spinel powder, or their introduction during the fabrication process to form the crushable spinel preform, can disturb the stoichiometric state achieved in the final preform, and thereby sharply curtail the ability of the final spinel insulated cable  
25 to withstand radiation and temperature stresses. The fabrication process described herein illustrates how this is accomplished.

In order for the finished crushable preform to  
30 possess a desired stoichiometric state that imparts superior stability to the metal sheathed cable, both the impurity levels and the stoichiometric state must be controlled. The procedure described herein accomplishes this.

The chemical makeup and method used for constructing  $\text{MgAl}_2\text{O}_4$  preforms 10 is as follows.

Preparation of Binder Solution

5        A binder solution is combined with a  $\text{MgAl}_2\text{O}_4$  homogenous powder to form a paste that is suitable for extruding and will substantially maintain the extruded shape. Accordingly, a first step involves the preparation of the binder solution.

10

The following list of items should be combined in the order listed and then heated to approximately 85°C. Substantial stirring is preferred during the heating process.

15

Triple Distilled Water, 3001 grams  
Glycerine, 567.5 grams  
Maycon-10, 56.75 grams  
Triton X-100, 5.7 grams

20

When the solution reaches 85°C, it should be removed from the heat, and 567.5 grams of Methocel #A4C should be stirred into the solution slowly. After approximately two minutes of stirring the solution appears milky in color. Thereafter, 1800 grams of triple distilled water are stirred into the milky solution. Preferably, the triple distilled water should be at approximately room temperature. This entire solution is preferably covered and aged overnight. Shelf life of the solution is estimated to be approximately six to eight weeks if kept cool (i.e., 7 to 10°C).

25

30

$\text{MgO-Al}_2\text{O}_3$  1:1 Stoichiometry Conformation

Before combining the binder solution with the spinel powder (available from TAM Ceramics, Inc. located at  
35    Niagara Falls, N.Y. 14305), as discussed above, it is preferable to examine the powder to ensure that

substantially all of the MgO has reacted with all of the  $\text{Al}_2\text{O}_3$  to form substantially pure  $\text{MgAl}_2\text{O}_4$ .

5 A homogeneous sample is taken from the powder using a "thief" tube or similar sampling device. The sample is then X-rayed using X-ray diffraction to determine if all of the powder is in an approximately 1:1 molar ratio of  $\text{Al}_2\text{O}_3$  to MgO to form the required  $\text{MgAl}_2\text{O}_4$  spinel compound. If the material is pure but the reaction is incomplete, 10 the powder is recalcined by placing it in a magnesium oxide crucible and heating the powder to a minimum of 1200°C in an air atmosphere in an electrically heated type firing furnace. Preferably, the 1200°C temperature is maintained for a four hour holding period. Thereafter, 15 the furnace is turned off and the recalcined powder is allowed to cool in the furnace. The recalcined powder is then broken up using an alumina mortar and pestle and ball milled using a conventional polypropylene lined ball mill and alumina balls or cylinders in dehydrated 20 methanol. Preferably, the recalcined powder is ball milled to a particle size of 1 to 10 microns.

After air drying at 125°C maximum temperature, the powder is again examined by X-ray diffraction to assure 25 an approximately 1:1 molar ratio of MgO to  $\text{Al}_2\text{O}_3$ , and that all of the material has been reacted.

#### Preparation of Extrusion Paste

30 With the binder solution and pure  $\text{MgAl}_2\text{O}_4$  ceramic powder prepared, they are combined to form an extrusion paste. Approximately 1000 grams of the  $\text{MgAl}_2\text{O}_4$  powder and 310 grams of the binder solution are added to a sigma blade type stainless steel mixer, such as is available from Paul O. Abbe, Inc., of Little Falls, New Jersey. 35 Preferably the total working chamber, including all moving parts, of the mixer should be coated with titanium

nitride. A titanium nitride coated cover plate of the mixer should be locked in place to prevent spillage.

5       The mixer is first operated until the contents appear uniform (approximately three minutes). At that time, 40 grams of a mixture of a 10% binder solution dissolved in 90% triple distilled water is added to the contents of the mixer. The resulting paste should be sufficiently stiff to have no deformation of the hole or  
10       outer diameter of the preform 10 after extrusion or drying.

15       After all of the ceramic powder has been added and mixed for 3 minutes, the batch is divided in half, and each half batch is mixed for approximately three to four minutes. Each half batch is then placed in a plastic bag and aged from an approximate minimum time of ten hours at a temperature of 7 to 10°C.

20       At this time, it is desirable to check the moisture content of the paste. Preferably, the moisture content should be approximately  $27 \pm 1$  percent. The moisture content can be determined either by using a weight loss method or by using a moisture balance, such as a Mettler  
25       LP16-M Moisture Determination System available from Fisher Scientific Co. of St. Louis, Missouri, as item number 01-913-93B. The weight loss method of determining moisture content involves accurately weighing the paste before and after drying at about 100°C for about four  
30       hours. The difference in weight corresponds to the amount of moisture lost therefrom.

      The paste should preferably be extruded within one week after preparation.

### Extrusion

The paste should be kneaded by hand on a clean plexiglass sheet for approximately two minutes to give it uniform plasticity and to form it into a shape for loading into an extruder. The extrusion process begins using, for example, a 40-ton extruder, such as is available from Loomis Products, Co. of Levittown, Pennsylvania. Preferably, the extruder has an extrusion chamber inner lining and ram plunger end plate of tungsten carbide.

With the extruder turned on, its ram plunger should be moved downward slowly into the extrusion chamber with the vacuum pressure also turned on. When the gauge begins to show ram pressure, the ram plunger should be stopped and held in place while the vacuum is operated for approximately four minutes before further operation of the extruder.

It should be appreciated that the extruder includes a pin used to form the opening in the preform 10 so that the wire 12 may be threaded therethrough. This pin is adjustable and should preferably be properly positioned or centered prior to actually extruding the paste.

Extrusion pressure should be held at about three to four tons, as registered on the pressure gauge. The resulting extrusions are preferably placed on 1/4" wide V shaped grooves in a plaster board (6" wide x 30" long x 3/4" total thickness) that has been dusted previously with GP-38 graphite powder, available from Union Carbide Coatings Corp. of Danbury, Connecticut.

### Drying

During the extrusion process, the damp extrusions should preferably be placed in a drying chamber. The



extrusions are preferably dried on the graphite dusted plaster boards at approximately 100°F maximum in a closed chamber for a minimum of approximately 25 hours. This drying is supplemented by overnight drying at 125 to 150°C in an air circulating dryer.

#### Cutting

After drying, the extrusions have a wood-like consistency and can be readily cut to a desired length for firing. For example the extrusions can be cut to a desired firing tile length using a back and top block, straight edge, and a safety-type stainless steel razor blade or Ex-acto® knife.

#### Loading

The cut extrusions should be loaded onto a mullite grain bonded hi-alumina type V-grooved setter tray, such as is available from Applied Ceramics, Inc., of Atlanta, Georgia, as part number TAC 218. Preferably, the V-grooves should be filled to approximately 1/8" over their depth with a plurality of the extrusions stacked therein.

Preferably the loaded hi-alumina grooved setter trays are stacked four high and two wide into a periodic furnace, such as a 3000 series furnace with Kanthal Super 33 heating elements, available from CM Furnaces, Inc. of Bloomfield, N.J.

#### Firing

The spinel rods are fired in an air atmosphere on the mullite bonded alumina refractory "V" trays. The furnace is heated to 400°C over a three hour period and held at that temperature for one hour. Thereafter, the temperature of the furnace is first raised to 1275°C at a rate of 150°C per hour, and then at 100°C per hour to a peak temperature of 1400°C. This peak temperature is held

for two hours, and then the heating elements of the furnace are turned off and the parts are allowed to cool overnight (minimum) in the closed furnace.

5       The spinel preforms 10 are now ready to be cut to specified length, usually two inches. The preforms 10 are preferably cut to a flat end cut using a back and top block straight edge and a four inch diameter diamond impregnated 1/32" wide cutting wheel.

10

#### Physical Tests

      To ensure that the preforms 10 are of suitable quality, several tests are performed. The modulus of rupture (MOR) is measured using a Chatillon model DFGHS-15 100 digital compressive force gauge on a model LTCM-4 mechanical test stand. The test stand is preferably equipped with a controlled motorized lifting table and a tungsten carbide base block having its tungsten carbide chisel edge tip positioned on the bottom of the digital force gauge. Preferably, the MOR of the preforms 10 20 should be approximately  $2000 \pm 200$  PSI.

      The outer diameter of the preforms 10 should be measured with, for example, a spring loaded electronic 25 digital read-out micrometer. Preferably, the outer diameter tolerance of the preforms 10 is approximately  $\pm 1.6\%$  for an outer diameter of about 0.200 inches.

      The inner diameter of the openings should be 30 measured using, for example, spring steel gauge pins accurate to  $\pm 0.0001$  inch. The gauge pins are available in intervals of 0.001 inch.

      Also, camber is measured and preferably should be 35 within the following tolerance:

      Typical camber: 0.005 inch/inch or mm/mm;

The specific method described above results in a preform 10 that is spinel ( $\text{MgAl}_2\text{O}_4$ ) of a substantially 1:1 molar ratio. In certain applications, however, it is preferable that a slight excess of  $\text{MgO}$  or  $\text{Al}_2\text{O}_3$  be present in the spinel compound.

The instant invention also finds beneficial applications in other swageable components or internal heater or thermocouple parts, such as spacers, inserts, mandrels, end seal inserts, and other internal parts used in cable and component design.

The ceramic insulator 10 has been described above as being composed exclusively of spinel. However, it is envisioned that the advantages of spinel insulation may be obtained through the use of a mixture that is comprised only partially of spinel. That is, spinel may be combined with other materials to produce a ceramic that exhibits comparable qualities to that of spinel alone. In particular, refractory dielectrics are expected to work well in combination with spinel. The term refractory dielectric is generally defined as a high temperature (or heat resistant) electrical insulator, and generally includes mineral compounds such as oxides, nitrides, etc., or, more particularly, silica ( $\text{SiO}_2$ ), kaolin, pyrophyllite, bentonite, talc, or other similarly good dielectric insulator material, such as  $\text{CeO}_2$ ,  $\text{Li}_2\text{O}$ ,  $\text{Li}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Sm}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{TiN}$ ,  $\text{Si}_3\text{N}_4$ ,  $\text{BaTiO}_2$ ,  $\text{Y}_2\text{O}_3$ ,  $\text{MgAl}_2\text{O}_4$ , and the like.

The refractory dielectrics have dielectric properties that approximates that of spinel, and, therefore, do not reduce the desirable electrical properties of spinel. That is, a spinel-refractory dielectric cable will retain, or at least approximate, the electrical performance of spinel alone.

Although particular detailed embodiments of the apparatus have been described herein, it should be understood that the invention is not restricted to the details of the preferred embodiment. Many changes in  
5 design, configuration, composition, and dimensions are possible without departing from the spirit and scope of the instant invention.

CLAIMS

1. Ceramic insulation for use in a mineral insulated electrical component, comprising spinel.

5

2. Ceramic insulation, as set forth in claim 1, including a refractory dielectric combined with said spinel, wherein said spinel forms at least about 51% by weight of the insulator.

10

3. Ceramic insulation, as set forth in claim 2, wherein said refractory dielectric is at least one of the group of silica ( $\text{SiO}_2$ ),  $\text{MgO}$ ,  $\text{Al}_2\text{O}_3$ , kaolin, pyrophyllite, bentonite, and talc.

15

4. Ceramic insulation, as set forth in claim 1, wherein said spinel contains about 28% of magnesia and about 72% alumina by weight.

20

5. Ceramic insulation, as set forth in claim 1, wherein said spinel contains a molar ratio of magnesium oxide to aluminum oxide of about 1:1.

25

6. Ceramic insulation, as set forth in claim 1, wherein said insulation is a swageable ceramic preform.

30

7. A paste suitable for firing into a ceramic for use as insulation in a mineral insulated electrical component, comprising a mixture of spinel and a binder solution.

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8. A paste, as set forth in claim 7, including a refractory dielectric combined with said spinel and binder solution, wherein said spinel forms at least about 51% by weight of the insulator.

5

9. A paste, as set forth in claim 8, wherein said refractory dielectric is at least one of the group of silica ( $\text{SiO}_2$ ),  $\text{MgO}$ ,  $\text{Al}_2\text{O}_3$ , kaolin, pyrophyllite, bentonite, and talc.

10

10. A paste, as set forth in claim 7, wherein said binder solution includes distilled water, methocel, and glycerin.

15

11. A paste, as set forth in claim 7, wherein said spinel includes magnesia and alumina.

20

12. A paste, as set forth in claim 7, wherein said spinel has a molar ratio of  $\text{MgO}$  to  $\text{Al}_2\text{O}_3$  of about 1:1.

25

13. A method for forming a ceramic to be used as insulation in a mineral insulated electrical component, comprising the steps of:

30

preparing a binder solution;

preparing a spinel powder;

35

combining said spinel powder and binder solution into a paste; and

firing said paste.

14. A method, as set forth in claim 13, wherein  
5 said step of preparing a binder solution includes the  
step of:

combining distilled water, methocel, glycerin,  
and a wetting agent.

10

15. A method, as set forth in claim 13, wherein  
said step of firing said paste includes the steps of:

15 heating said paste in an air atmosphere at a  
first preselected temperature and for a first preselected  
period of time sufficient to substantial burn off said  
binder solution; and

20 heating said extruded paste in an air  
atmosphere at a second, higher preselected temperature  
and for a second preselected period of time.

25 16. A method, as set forth in claim 13, including  
the step of forming said fired ceramic into granulated  
powder.

30 17. A mineral insulated electrical component,  
comprising:

a conductive wire;

35 a sheath positioned about and spaced from said  
conductive wire; and

ceramic insulation positioned between said conductive wire and said sheath, said insulation being comprised of spinel.

5

18. A mineral insulated electrical component, as set forth in claim 17, wherein said spinel includes magnesia and alumina.

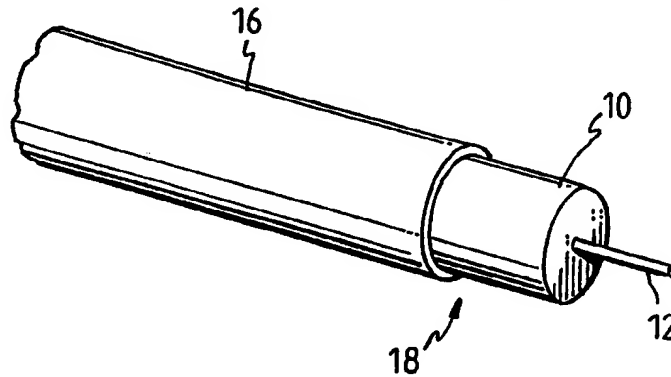
10

19. A mineral insulated cable, as set forth in claim 17, wherein said spinel contains a molar ratio of magnesium oxide to aluminum oxide of about 1:1.

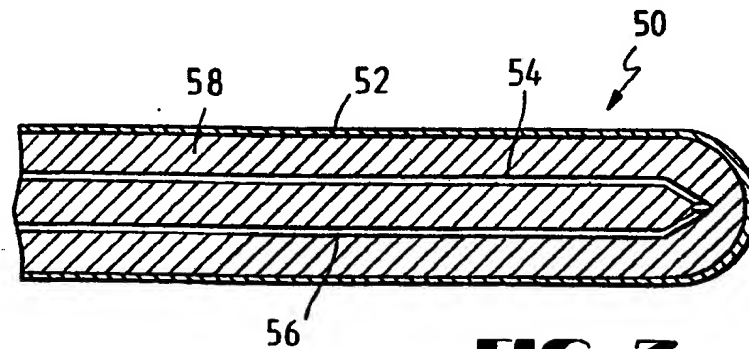
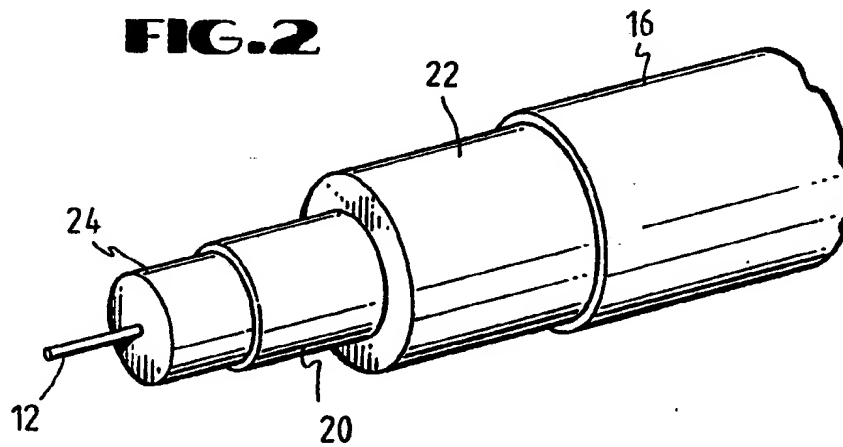


1/1

**FIG.1**



**FIG.2**



**FIG.3**

# INTERNATIONAL SEARCH REPORT

International Application No.

PCT/US 92/07456

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (if several classification symbols apply, indicate all) <sup>4</sup> According to International Patent Classification (IPC) or to both National Classification and IPC IPC <sup>5</sup> : H 01 B 3/12, H 01 B 13/06, G 01 K 7/02, G 21 C 17/00														
<b>II. FIELDS SEARCHED</b> <div style="text-align: center; border-top: 1px solid black; border-bottom: 1px solid black;">Minimum Documentation Searched <sup>7</sup></div> <table style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 25%; border: 1px solid black; text-align: left;">Classification System</th> <th style="border: 1px solid black; text-align: left;">Classification Symbols</th> </tr> <tr> <td style="border: 1px solid black; vertical-align: top;">IPC<sup>5</sup></td> <td style="border: 1px solid black; vertical-align: top;">H 01 B, H 05 B 3/00, G 01 K 7/00, G 21 C 17/00, G 01 T 7/00</td> </tr> </table> <div style="text-align: center; border-top: 1px solid black; border-bottom: 1px solid black;">Documentation Searched other than Minimum Documentation to the extent that such Documents are included in the Fields Searched <sup>8</sup></div>			Classification System	Classification Symbols	IPC <sup>5</sup>	H 01 B, H 05 B 3/00, G 01 K 7/00, G 21 C 17/00, G 01 T 7/00								
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IPC <sup>5</sup>	H 01 B, H 05 B 3/00, G 01 K 7/00, G 21 C 17/00, G 01 T 7/00													
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT <sup>9</sup></b> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 10%; border: 1px solid black;">Category <sup>9</sup></th> <th style="width: 60%; border: 1px solid black;">Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup></th> <th style="width: 30%; border: 1px solid black;">Relevant to Claim No. <sup>13</sup></th> </tr> <tr> <td style="border: 1px solid black; vertical-align: top;">X;Y</td> <td style="border: 1px solid black; vertical-align: top;">           JP, A, 60-214 295            (TOSHIBA) 26 October 1985            (26.10.85),            see claims; page 3, fig. 2.            &amp; Chemical Abstracts,            Columbus, Ohio, USA,            vol. 104, no. 26, issued            June 30, 1986; page 512,            the abstract-no. 232 694y            --         </td> <td style="border: 1px solid black; vertical-align: top; text-align: center;">           1, 4, 5,            17-19;            7, 13,            15         </td> </tr> <tr> <td style="border: 1px solid black; vertical-align: top;">Y</td> <td style="border: 1px solid black; vertical-align: top;">           DE, C, 839 664            (SIEMENS-SCHUCKERTWERKE AG)            23 May 1952 (23.05.52),            see claims; page 2,            lines 9-70.            --         </td> <td style="border: 1px solid black; vertical-align: top; text-align: center;">           7, 13,            15         </td> </tr> <tr> <td style="border: 1px solid black; vertical-align: top;">X</td> <td style="border: 1px solid black; vertical-align: top;">           US, A, 4 200 077            (KAUHL et al.) 29 April 1980            (29.04.80),            see claims; column 2, line 18            - column 3, line 10; fig.            --         </td> <td style="border: 1px solid black; vertical-align: top; text-align: center;">           1-6;            17-19         </td> </tr> </table>			Category <sup>9</sup>	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No. <sup>13</sup>	X;Y	JP, A, 60-214 295 (TOSHIBA) 26 October 1985 (26.10.85), see claims; page 3, fig. 2. & Chemical Abstracts, Columbus, Ohio, USA, vol. 104, no. 26, issued June 30, 1986; page 512, the abstract-no. 232 694y --	1, 4, 5, 17-19; 7, 13, 15	Y	DE, C, 839 664 (SIEMENS-SCHUCKERTWERKE AG) 23 May 1952 (23.05.52), see claims; page 2, lines 9-70. --	7, 13, 15	X	US, A, 4 200 077 (KAUHL et al.) 29 April 1980 (29.04.80), see claims; column 2, line 18 - column 3, line 10; fig. --	1-6; 17-19
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<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p><sup>14</sup> Special categories of cited documents: <sup>14</sup></p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"Z" document member of the same patent family</p> </div> </div>														
<b>IV. CERTIFICATION</b> <table style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 50%; border: 1px solid black; vertical-align: top;">           Date of the Actual Completion of the International Search   <div style="text-align: center;">09 November 1992</div> </td> <td style="width: 50%; border: 1px solid black; vertical-align: top;">           Date of Mailing of this International Search Report   <div style="text-align: center;">20 NOV 1992</div> </td> </tr> <tr> <td style="border: 1px solid black; vertical-align: top;">           International Searching Authority   <div style="text-align: center;">EUROPEAN PATENT OFFICE</div> </td> <td style="border: 1px solid black; vertical-align: top;">           Signature of Authorized Officer   <div style="text-align: center;">KUTZELNIGG e.h.</div> </td> </tr> </table>			Date of the Actual Completion of the International Search  <div style="text-align: center;">09 November 1992</div>	Date of Mailing of this International Search Report  <div style="text-align: center;">20 NOV 1992</div>	International Searching Authority  <div style="text-align: center;">EUROPEAN PATENT OFFICE</div>	Signature of Authorized Officer  <div style="text-align: center;">KUTZELNIGG e.h.</div>								
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III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, " with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	<p>WO, A1, 90/14 671  (METAL MANUFACTURES LIMITED)  29 November 1990 (29.11.90),  see page 5, lines 1-26.  -----</p>	<p>7,10,  13-15</p>

# ANHANG

zum internationalen Recherchen-  
bericht über die internationale  
Patentanmeldung Nr.

# ANNEX

to the International Search  
Report to the International Patent  
Application No.

# ANNEXE

au rapport de recherche inter-  
national relatif à la demande de brevet  
international n°

PCT/US 92/07456 SAE 64611

In diesem Anhang sind die Mitglieder  
der Patentfamilien der im obenge-  
nannten internationalen Recherchenbericht  
angeführten Patentdokumente angegeben.  
Diese Angaben dienen nur zur Unter-  
richtung und erfolgen ohne Gewähr.

This Annex lists the patent family  
members relating to the patent documents  
cited in the above-mentioned inter-  
national search report. The Office is  
in no way liable for these particulars  
which are given merely for the purpose  
of information.

La présente annexe indique les  
membres de la famille de brevets  
relatifs aux documents de brevets cités  
dans le rapport de recherche inter-  
national visée ci-dessus. Les renseigne-  
ments fournis sont donnés à titre indica-  
tif et n'engagent pas la responsabilité  
de l'Office.

Im Recherchenbericht angeführtes Patentdokument Patent document cited in search report Document de brevet cité dans le rapport de recherche	Datum der Veröffentlichung Publication date Date de publication	Mitglied(er) der Patentfamilie Patent family member(s) Membre(s) de la famille de brevets	Datum der Veröffentlichung Publication date Date de publication
JF A2 60214295	26-10-85	keine - none - rien	
DE 839664		keine - none - rien	
US A 4200077	29-04-80	BE A1 871246 BR A 7806773 DE A1 2746595 FR A1 2406089 GB A1 2006334 GB B2 2006334 JP A2 54065227	01-02-79 08-05-79 26-04-79 11-05-79 02-05-79 03-03-82 25-05-79
WO A1 9014671	29-11-90	AU A1 56608/90 AU B2 626683 CA AA 2055478 EP A1 472540 EP A4 472540	18-12-90 06-08-92 18-11-90 04-03-92 03-06-92